Article ID: 1004 - 6326(1999)03 - 0451 - 06

Continuous recrystallization in pure Al-1.3 % Mn investigated by local orientation analysis①

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Abstract: Local orientation analysis was used to investigate the continuous recrystallization process in a pure Al-1.3 % Mn alloy with the emphasis on the influence of matrix orientations on the subgrain growth and precipitation. Results show that the differences of (mis) orientations in deformed matrices give rise to inhomogeneous subgrain growth and precipitation with respect to precipitate density and morphologies. Moreover, no apparent high angle grain boundaries were developed by accumulation of misorientations during subgrain growth.

Key words: Al Mn; continuous recrystallization; precipitation; ODF; texture **Document code: A**

1 INTRODUCTION

The classical works on continuous recrystallization (or recrystallization in situ or extended recovery) are those of Hornbogen and Koster[1~3] on Al-Fe and Al-Cu. It is taken for granted that the Zener pinning due to secondary precipitation is the main reason for the occurrence of continuous recrystallization. The rearrangement of dislocations and the subgrain growth are controlled by the coarsening of large precipitates and dissolving of fine precipitates. This process is generally considered to be homogeneous in the whole sample. However, some recent researches indicate that continuous recrystallization can be in different forms. It was suggested[4] that during recovery in pure Al the formation of high angle grain boundaries as well as the structure reconstruction can be obtained by an accumulation of small misorientation during subgrain growth. The work on 1xxx Al alloy[5,6] indicated, on the other hand, that high angle grain boundaries did not form during annealing by subgrain growth, but existed already before annealing.

Despite some existed investigations on continuous recrystallization in Al-Mn (3xxx series) alloys[7~10], most of them were focused on the microstructural observation and macrotextural measurements due to the limitation of experimental techniques and the microtextural information is still not well known. This work intends to inspect the continuous recrystallization under the influence of matrix orientation by local orientation measurements.

2 EXPERIMENTAL

High purity Al with 1.3 % Mn (mass fraction) was subject to solution treatment for 5 h at 630 ℃ and then exposed to a series of three-axial forgings and recrystallization annealings to minimize the influence of a possibly existing initial texture and to reduce the initial grain size to 120 μm. The samples were cold rolled to thickness reductions of 80 % ~ 97 % and isothermally annealed at 350 ℃ in a salt bath. Microhardness measurement and structural observation con-

① Project 97 - 832 supported by the Science Foundation of Education Ministry of China for Returned Overseas Scholars
Received Aug. 9, 1998; accepted Nov. 10, 1998

firmed that continuous recrystallization prevailed below 350 ℃. Macrotextures were determined by X-ray diffraction and orientation distribution functions (ODF) were calculated according to the series expansion method. Local orientations were obtained by EBSD in a SEM[11]. Gauss ODFs were calculated by associating each single orientation with a Gauss type peak with a scatter width of 5°.

3 RESULTS AND ANALYSES

3.1 Macrotexture

Fig.1 illustrates the macrotexture measured on an 80 % rolled, 350 ℃ for 34 d annealed sample, indicating retained rolling textures.

Fig.1 Macrotextures in continuously recrystallized samples
(80 % rolling; 350 ℃, 34 d)

3.2 Subgrain growth

3.2.1 Microstructural evolution and its inhomogeneities

Despite the gradual subgrain growth on macroscale, two types of inhomogeneities were observed in microscale. One is the inhomogeneity in subgrain size, i.e., even in the absence of precipitates the subgrain growth was inhomogeneous. Subgrains may be larger than 10 μm or smaller than 1 μm in size. The other is the inhomogeneity in precipitate density (Fig.2) indicating that, despite the large difference of precipitate density in the same TEM foil, the subgrain size in both micrographs were similar. These phenomena reveal the influence of misorientation: The misorientation in regions of Fig.2(a) was large and thus the precipitation proceeded quickly, which in turn inhibited the subgrain growth, whereas the misorientation in Fig.2(b) was small and the retarding forces originated from the small mobility of the subgrain boundary and segregated Mn atoms, thus the subgrain sizes in the two regions were similar.

3.2.2 Misorientation evolution

The evolution of the misorientations of neighbouring subgrains during annealing were measured by orientation scanning along TD and RD at rolling plane of 80 % and 97 % rolled

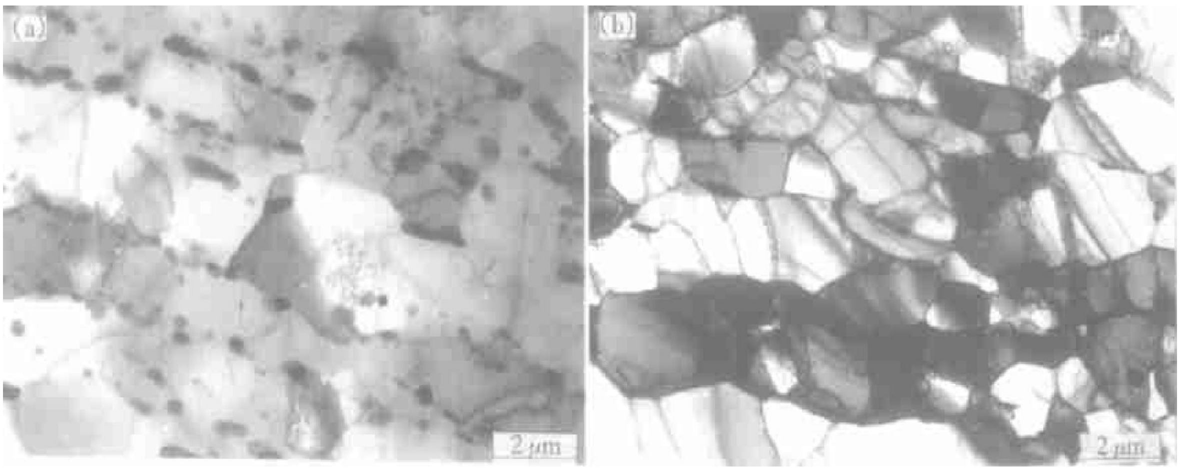

Fig.2 An example of inhomogeneity during subgrain growth
(86 %rolled; 350 ℃, 9.5 d)

samples. Fig.3 shows an example in the 80% rolled sample. The softening process was followed by referring to the microhardness value, the sharpness of EBSD patterns and by the TEM observations, demonstrating that, at the time when the microhardness decreased nearly to the value of the discontinuously recrystallized samples, there were virtually no new high angle grain boundaries developed during annealing, only prior grain boundaries were detected. Although more high angle grain boundaries were found in 97% rolled samples than in 80% ones, they were mainly either prior grain boundaries or had formed during deformation. This conclusion is based on the fact that, if high angle grain boundaries were formed by the accumulation of misorientations of subgrains during subgrain growth, they should be in the range of 15° ~ 25° but not remarkably larger than this value.

Fig.3 Evolution of misorientations in 80% rolled, 350 ℃ annealed samples

3.3 Influence of matrix orientation on precipitation

3.3.1 At early stage of annealing

Two regions with different precipitate densities were observed. In some deformed bands precipitation occurred quickly, whereas there were hardly any precipitates in other deformed bands. Orientation measurements revealed differences in orientation and misorientation (Fig.4) demonstrating that not only the misorientations in the region with more precipitates were larger than those in the region with less precipitates (Fig.4(a)), but also that such regions had more scattered rolling orientations (less B{110}〈112〉 orientations, Fig.4(b)) with respect to the exact rolling texture orientations in the region without precipitates (Fig.4(c)). Thus the high percentage of large misorientations in Fig.4(a) was certainly caused to some extent by prior grain boundaries. So it can be concluded that, because of the small misorientations in the deformed bands with typical rolling orientations, precipitation was delayed.

Fig.4 Statistic determination of (mis) orientations in two regions

(a) —Distribution of misorientations in two types of regions;
(b) —Orientations of region with more precipitates;
(c) —Orientations of region with less precipitates

3.3.2 At a later stage of annealing

As annealing proceeded, precipitation occurred gradually in the regions free from precipitates at the early stage of annealing and thus, the precipitates distributed relatively homogeneously in the matrix. However, precipitates formed at different periods had different shapes, being either round or elongated, and were distributed in differently oriented matrices, as shown in Fig.5, illustrating larger misorientations in the region with round precipitates and smaller misorientations in the region with elongated precipitates. In Fig.5(a) orientation scanning was performed at a length of 60 μm in TD on the rolling plane indicating a single B oriented grain. In Fig.5(b), an orientation scanning revealed large misorientations, only one grain boundary can be seen on the micrograph.

To obtain statistical information about the influence of the matrix orientation on the precipitate's shapes, the orientations of these two types of regions were measured and presented in Fig.6, confirming that elongated precipitates were mainly present in the B matrix with small misorientations, whereas round precipitates were mainly concentrated in the C{112}⟨111⟩/S{123}⟨634⟩ matrix with large misorientations. This demonstrates that the difference in precipitate's shapes was related with the misorientation and, essentially, with the matrix orientation. It should be noted that Fig.6 is not similar to Fig.4, because the elongated precipitates occurred mainly in the B bands and not in the matrix with the exact rolling texture orientations.

The subgrain sizes in the two regions with different shapes of precipitates were estimated by EBSD orientation scanning in the rolling plane of the 80% rolled, 350 ℃, 34 d annealed specimen. The determination of the subgrain size was not based on morphological observation, but the change of the EBSD pattern. The average subgrain size in the region with round precipitates was 5.9 μm (143 subgrains) and 7.2 μm in the region with elongated precipitates (130 subgrains). The same tendency was obtained when the subgrain size was classified according to the matrix orientation (B or C/S). This was in accordance with the precipitate densities of the two regions. Since all elongated precipitates had similar arrangements locally (Fig. 5(a)), they should have special crystallographic orientation relationships with the matrix and this is discussed in a separate paper[12].

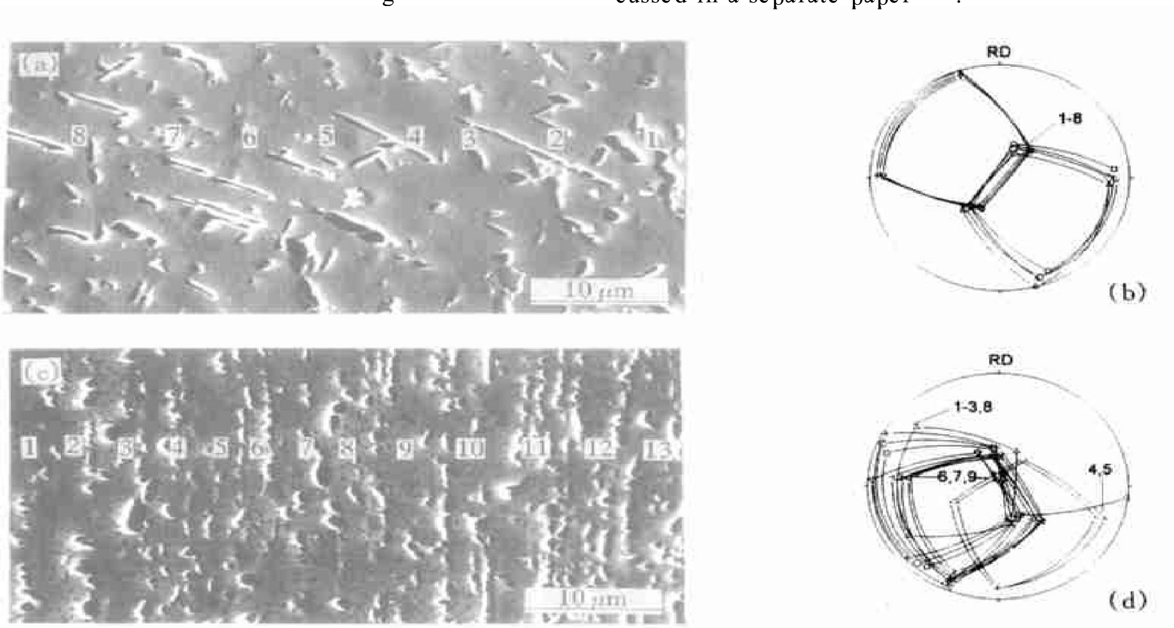

Fig.5 Example of precipitation matrix at late stage of annealing (80% rolling; 350 ℃, 34 d)

(a), (b) — Micrograph and orientation changes in region with elongated precipitates;
(c), (d) — Micrograph and orientation changes in region with round precipitates

Fig.6 Statistic determination of orientations in two regions
(80 % rolling; 350 ℃, 34 d)
(a) —Orientations of regions with elongated precipitates;
(b) —Orientations of regions with round precipitates

4 DISCUSSION

4.1 Traditional model of continuous recrystallization

The misorientation evolution in Fig.3 indicates only a small chance for the formation of high angle grain boundaries by subgrain growth. This can be understood in another way: The misorientation in B oriented bands was generally small. On the two sides of a B oriented band (with a width of over 20 μm) along TD the maximal misorientation measured is less than 15° (Fig.5a). So, it is impossible that a new high angle grain boundary could form by the accumulation of misorientations during subgrain growth in this region. Furthermore, owing to the pinning of precipitates, subgrains can not reach such size (the mean subgrain size is 7.2 μm). Similarly, there are also a lot of regions in the C/S matrices with small and homogeneous misorientations (Fig.5(c)), where no large angle grain boundaries can form because of the precipitate pinning (the mean subgrain size is 5.9 μm). Since the misorientations in many deformed grains are small, statistically high angle grain boundaries should not form by subgrain growth but exist before annealing as suggested in Ref.[5,6].

4.2 Difference of misorientations in deformed matrices with different orientations

Results in Figs.4 and 6 clearly reveal the difference of misorientations in deformed matrices with different orientations. This is in accordance with the investigation on compressed single crystals Al with B, S and C orientations[13]. The significance of this fact is that large strain in polycrystalline materials always leads to the formation of the so called β fibre composing of C, S and B orientations and thus inhomogenous misorientation distribution. Such difference in misorientations certainly exerts strong influence on recrystallization process. For instances, during discontinuous recrystallization, smaller misorientations in the B matrix result in the formation of the B/R component, which competes strongly with the cube texture formed in S/C matrices with larger misorientations[14]; at the presence of large particles, PSN (particle stimulated nucleation) would take place faster in C/S matrices than in B matrix[15]; while annealing at low temperatures, the smaller misorientations in B matrix would give rise to the postponed precipitation and moreover the morphological transition from round to elongated ones. The increase of strain and the decrease of initial grain size will reduce the influence of matrix orientations.

5 CONCLUSIONS

(1) Although the subgrain growth was gradual on the macroscale and was controlled by precipitation, on the microscale the subgrain size and distribution of the precipitates were inhomogeneous. This indicates the influences like Mn atoms in solution and matrix orientation.

(2) Normally, high angle grain boundaries did not develop by subgrain growth.

(3) Rolling always leads to the formation of a β fibre comprising B, S and C orientations. The misorientations in the matrices of these different components are systematically different. Misorientations are larger in the C/S matrix than in the B matrix, giving rise to a difference in precipitation with respect to time and shape: At

the early stages of annealing two regions with clearly different precipitate densities were observed, namely the scattered rolling orientation matrix with a lot of precipitates, and the typical rolling orientation matrix, free from precipitates. At later stages two regions with different shapes of precipitates were observed, i.e. round precipitates mainly in the C/S matrix and elongated precipitates in the B matrix.

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(Edited by Peng Chaoqun)